

Experiment Report Form

The double page inside this form is to be filled in by all users or groups of users who have had access to beam time for measurements at the ESRF.

Once completed, the report should be submitted electronically to the User Office via the User Portal:

<https://www.esrf.fr/misapps/SMISWebClient/protected/welcome.do>

Reports supporting requests for additional beam time

Reports can be submitted independently of new proposals – it is necessary simply to indicate the number of the report(s) supporting a new proposal on the proposal form.

The Review Committees reserve the right to reject new proposals from groups who have not reported on the use of beam time allocated previously.

Reports on experiments relating to long term projects

Proposers awarded beam time for a long term project are required to submit an interim report at the end of each year, irrespective of the number of shifts of beam time they have used.

Published papers

All users must give proper credit to ESRF staff members and proper mention to ESRF facilities which were essential for the results described in any ensuing publication. Further, they are obliged to send to the Joint ESRF/ ILL library the complete reference and the abstract of all papers appearing in print, and resulting from the use of the ESRF.

Should you wish to make more general comments on the experiment, please note them on the User Evaluation Form, and send both the Report and the Evaluation Form to the User Office.

Deadlines for submission of Experimental Reports

- 1st March for experiments carried out up until June of the previous year;
- 1st September for experiments carried out up until January of the same year.

Instructions for preparing your Report

- fill in a separate form for each project or series of measurements.
- type your report, in English.
- include the reference number of the proposal to which the report refers.
- make sure that the text, tables and figures fit into the space available.
- if your work is published or is in press, you may prefer to paste in the abstract, and add full reference details. If the abstract is in a language other than English, please include an English translation.



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|---|--|--------------------------------------|
| | Experiment title: Influence of compressed gases on the structure of lipid monolayers | Experiment number: SI-2362 |
| Beamline: ID15A | Date of experiment: from: 31/10/12 to: 06/11/12 | Date of report: 20/02/2013 |
| Shifts: 18 | Local contact(s): Dr. Oleg Konovalov | <i>Received at ESRF:</i> |
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Report:

In the human body, cells and functional units are spatially limited by the cell membrane, a lipid bilayer with functional proteins. A lipid bilayer is a double layer consisting of amphiphilic molecules that order spontaneously, so that their hydrophilic head group points towards the aqueous cell matrix and the hydrophobic tail groups of two molecules are opponent. An intact cell membrane is essential for the correct cell functionality. Depending on their solubility and size, gas molecules can cross the cell barrier by diffusion through the membrane. Thus, the question of how diverse harmful substances can modify membranes is of eminent importance for the understanding of the basic mechanisms of malfunctions.

If one wants to reduce the complexity of the bilayer and gain access to more basic mechanisms, a well-established model system are simple lipid monolayers. These Langmuir layers are formed at the water-gas interface if certain lipids are applied carefully to the water surface. The head group is then oriented to the aqueous phase and the tail group is pointed to the gas phase.

We studied the pressure dependent effect of n-butane and octafluoropropane on the structure of stearic acid monolayers. Experiments were performed in the following way. 20ml of ultrapure water were filled into the experimental cell. 90 µl of a 1 mM solution of stearic acid in chloroform was applied carefully onto the water surface with a syringe. Reference measurements with a monolayer of stearic acid were recorded. Subsequently, the cell was flushed with the respective gas and pressurized. Then, a XRR and / or GID scan was performed. Because of the high flux at ID 10, only one measurement could be recorded on the sample, otherwise beam damage occurred. Thus, a fresh sample was prepared for each measurement.

To obtain an estimate of the systematic error caused by the sample preparation, the pressure steps had to be reproduced several times, which is very time consuming. Figure 1(a) shows the reflectivity of a pure water

interface, a water interface with a stearic acid monolayer, and the water/stearic acid system at an absolute n-butane pressure of 1.44 bar at $T = 10^\circ\text{C}$. The reflected intensity is plotted versus q_z^2 and is normalized by the Fresnel reflectivity. Figure 1(b) shows the electron density profiles, normalized by the water electron density, obtained from a fitting procedure. While the water interface does not show any layer formation, the lipid monolayer can be clearly distinguished with its head and tail group on the red curve. However, when the system is pressurized with an absolute pressure of 1.44 bar of n-butane, the electron density in the region of the head and tail group is altered. While the electron density of the head group is broadened, the electron density in the tail region drops down. Thus, the gas molecules prefer to accumulate between head and tail group. However this result has to be verified.

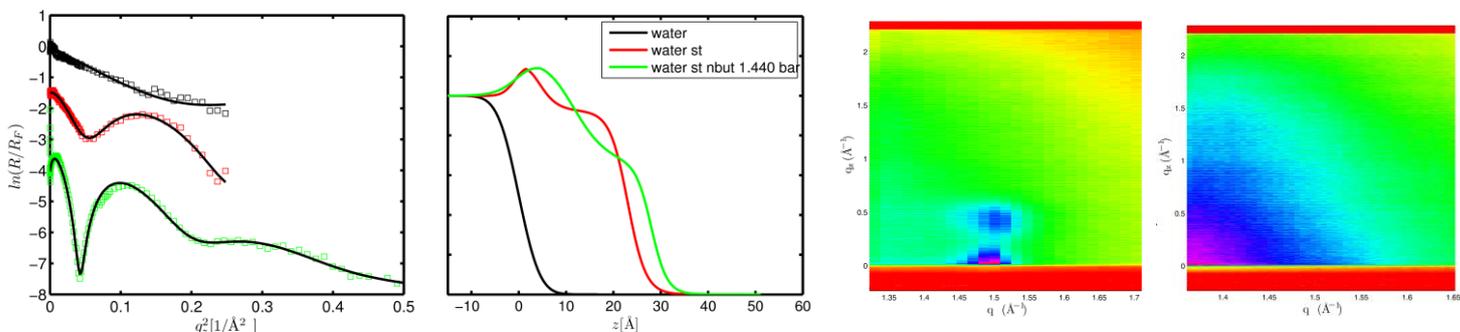


Figure 1(a) Reflectivity of water with lipid monolayer and n-butane. (b) Electron density along the vertical direction. (c) GID scan of a stearic acid monolayer. (d) GID scan of the system at 1.45 bar n-butane.

A GID scan on the stearic monolayer shows a crystalline lateral structure with slightly tilted tailgroups, detectable from the peak at $q_{\text{par}} \sim 1.5 \text{\AA}^{-1}$ in Figure 1(c). While peaks can still be observed at lower gas pressures, no peak is detectable on the GID scans at 1.45 bar of n-butane in Figure 1(d). Thus, the lateral structure of the Langmuir layer is more and more affected with increasing gas pressure.

Similar experiments were performed on a stearic acid monolayer in the presence of octafluoropropane (C_3F_8). From the GID scans in Figure 2(a) and (b), it is visible that the Bragg peak around $q_{\text{par}} \sim 1.45 \text{\AA}^{-1}$ disappears if the pressure is increased to an absolute pressure of 2 bar. This result reveals that the lateral order of the Langmuir layer is destroyed by the increasing pressure. A more detailed data analysis is currently in progress.

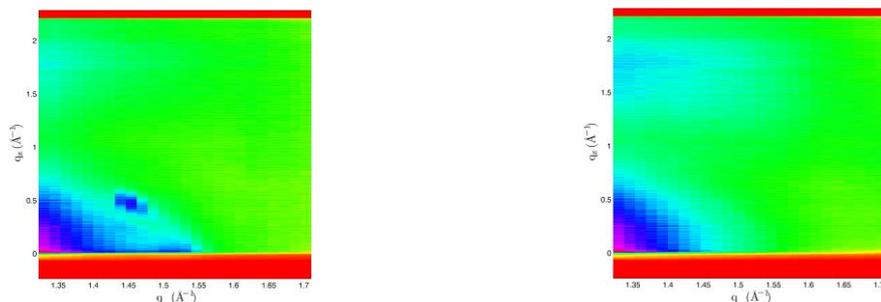


Figure 2 (a) GID scan of a stearic acid monolayer flushed with C_3F_8 . (b) At 2 bar of C_3F_8

In conclusion, these first measurements show the feasibility of XRR and GID on Langmuir layers under increased gas pressure. We detected a modification of the Langmuir monolayer structure and found that from a certain critical pressure on, the Langmuir layer collapses. The reflectivity measurements show that the gas molecules penetrate the Langmuir layer also at low gas pressures. They seem to accumulate between head and tail group of the lipids. This accumulation goes in hand with the destruction of lateral crystalline order. However, additional experiments are needed to verify the results and to investigate and understand in more detail the interaction mechanisms between gas and Langmuir layer, for example by using gases with a different radius of gyration.